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and Space Administration

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SUMMARY

A low-density (64 kg/m^3), polyimide foam material was tested in an arc tunnel to determine its potential for heat-shield application on aerospace vehicles. The results show that the material has some reuse potential at surface temperatures as high as 750 K (0.43 cm recession in 12 000 sec). When a black refractory paint was applied to the surface of the material, the surface recession was negligible at 750 K. An analytical thermal conductivity was derived for this material which, combined with measured thermal property values, can be used to make preliminary-design thickness calculations for heat-shield applications.

INTRODUCTION

Reusable heat shields which have good insulative properties are generally made from ceramic materials in low-density forms such as foams or sintered fibers. These low-density ceramic materials are very brittle and fragile, and considerable expense is required to incorporate them into reliable heat shields.

In some applications, large areas, such as the upper surface of the space shuttle orbiter, experience relatively moderate heating rates. High-temperature organic materials should have reuse capability in these areas. If these materials can be fabricated in low-density forms which retain flexibility and toughness, heat-shield design effort, complexity, cost, and weight should be reduced.

One class of high-temperature organic materials which has been extensively used is polyimide. The development of a low-density polyimide foam for container cushioning is described in reference 1. This foam is moldable, resilient, and nonabrading. Because of these desirable characteristics, a range of foam density was investigated for heat-shield application (ref. 2). The optimized, low-density (64 kg/m^3) polyimide foam was tested in an arc tunnel to determine its potential for heat-shield application. The results of the tests are described in this paper.

MATERIAL DEVELOPMENT

Fabrication of the material and determination of its physical characteristics are described in detail in reference 2. Basic material fabrication involved three steps.

First, a commercially available, foamable, polyimide powder was heated for approximately one-half hour at 448 K and then for one-half hour at 573 K. This procedure yielded very low-density (10 kg/m^3), irregular, tough foam. Next, this foam was shredded by a high-speed grinder to produce a bulky, lightweight, finely divided material having a density of about 8 kg/m^3. Finally, the shredded material was placed in a steel mold, compressed to the desired density, and heated for three hours at 600 K. This process resulted in a very fine textured, tough, resilient foam. The foam was prepared in six densities ranging from 32 to 128 kg/m^3. Some of the foam was postcured to 700 K. A series of thermal, mechanical, and environmental tests were performed on the foam.

As expected, the foam with the lowest density had the lowest thermal conductivity. However, the 32 and 48 kg/m^3 foams had marginal mechanical properties. The postcured foam had much better resistance to elevated temperature exposure. Therefore, postcured 64 kg/m^3 foam was selected as having the best combination of properties for heat-shield application and was used in this study.

ARC-TUNNEL TESTS

Test Specimens and Fixture

The foam test specimens were approximately 12.7 cm square and 2.54 cm thick. Details of a typical test specimen are shown in figure 1. The test specimens were mounted on the side of a water-cooled test fixture as shown in figure 2. The surface of the specimen was about 0.6 cm below a rearward-facing step. The heating-rate distribution over the surface of the specimen was more uniform with a rearward-facing step than when the specimen was mounted flush with the holder (ref. 3).

The surface of one test specimen was coated with the black refractory paint described in reference 4. Approximately 0.03 g/cm^3 of paint was applied to the specimen. The paint had a very low viscosity and did not appear to seal the surface.

Test Environments

The arc tunnel used for the tests is described in reference 3. The test environments used in this investigation are given in table I. The lowest heating rate, test condition 1, was established by the minimum operating capability of the tunnel.

The cold-wall heating rate was determined by the initial temperature-rise rate of a thin-skin calorimeter as described in reference 5. The calorimeter configuration is shown in figure 3. The local pressure at the surface was measured with a calibration model the same size and shape as the test specimens. The heating-rate and pressure values given in table I were obtained at the center point of the respective models.

Total enthalpy was determined by using the following established facility procedure. Probes were used to determine the stagnation heating rate and stagnation pressure in the center of the stream. A correlation equation was then used to calculate the enthalpy (ref. 6). All specimens, except one, were tested in an air environment. One specimen was tested in a nitrogen stream.

Test Procedure

The specimens were tested according to the matrix given in table II with each cycle lasting 1200 seconds. Specimen thickness was measured at the center point. The surface recession at the center point was approximately equal to the average recession of each specimen. Thickness and mass measurements were made only at the times shown; therefore, the measurements made after the fifth and tenth cycles were cumulative totals. For each test, tunnel operating conditions were established, and the test environment was allowed to stabilize. Heating-rate and pressure measurements were made. The specimen was inserted into the stream and exposed to the test environment for 1200 seconds. The specimen was removed from the stream, and heating-rate and pressure measurements were repeated. Heating-rate and pressure measurements made before and after each test were essentially the same. The 1200-second test time was determined by the arc-tunnel vacuum system capabilities.

RESULTS AND DISCUSSION

Thermogravimetric Analysis and Surface Emittance

A standard thermogravimetric analysis (TGA) apparatus was used to determine the temperature—mass-loss characteristics of the polyimide foam. The results, given in figure 4, show that mass loss was very small below a temperature of about 750 K.

The surface emittance of the polyimide foam was measured by using the method and apparatus described in reference 7, and the results are given in figure 5. The total emittance of the uncoated foam was 0.5. The total emittance of the coated specimen was 0.68.

Arc-Tunnel Tests

Test results are shown in table III and are plotted in figures 6 to 10. The tests did not produce any significant visible change in the surface color or texture of any of the specimens.

The recession and mass loss of specimens 1 to 6 are shown in figure 6 as functions of cold-wall heating rate and number of test cycles. The results show that, as expected, both recession and mass loss were greater for higher heating rates and more test cycles.

Note that at the lowest heating rate, the specimen receded only 0.43 cm after 10 cycles (12 000 sec) of testing.

The effect of testing in a nitrogen stream rather than in air is shown in figure 7. At a heating rate of 20.4 kW/m^2, the specimen tested in nitrogen receded about 40 percent less than the specimen tested in air. This difference in recession was probably caused by oxidation. The recession in the nitrogen stream was probably caused by mechanical erosion and/or shrinkage.

The effect of the surface coating is shown in figure 8. The coated specimen was tested for 10 cycles at condition 1 and then 5 cycles at condition 2. The coating essentially eliminated recession at both test conditions. The mass of the coated specimen at test condition 1 did not change after the first test cycle. At test condition 2, the mass loss was slightly greater after cycling.

The thickness-loss to mass-loss ratio is shown in figure 9 as a function of the cold-wall heating rate. For uncoated specimens, the ratio generally was larger as a function of both heating rate and number of test cycles. The reason for this effect was that, although the thickness of the thermally affected layer of the material remained approximately constant, the affected layer became a smaller part of the total recession as the total recession became larger. For the coated specimen, the ratio became smaller because all the thickness loss occurred during the first cycle and the thermally affected layer grew slightly during subsequent cycles.

The temperature rise at the back surface of each specimen, averaged over the test cycles, is plotted as a function of cold-wall heating rate in figure 10. The temperature rise at the two highest heating rates was not as great as expected, primarily because the large surface recession significantly increased the depth of the rearward facing step (fig. 2) and caused a decrease in the heating rate (ref. 3). The temperature rise of the coated specimen was significantly less than that of the uncoated specimens at the same test conditions, because the higher surface emittance resulted in a lower surface temperature and the small surface recession provided a longer conduction path.

Analytical Results

The numerical analysis of reference 8 was used to determine the thermal response of the polyimide foam to the various test environments. Because surface recession is not included, the analysis is more accurate at the lower heating rates. When the analysis was used at the higher heating-rate conditions, reductions in heating rate due to recession were not included, which compensates to some extent for omitting surface recession.

The variation of thermal conductivity with temperature which was used as input data for the analysis is shown in figure 11. This conductivity was obtained through a

trial and error procedure tempered by a knowledge of the probable temperature dependence (ref. 9). The calculated thermal conductivity agrees reasonably well with the limited conductivity data from reference 2. The value of specific heat used in the calculations was 1.13 J/g-K. The appropriate measured emittance value was used in each calculation.

A comparison of calculated with measured back-surface temperature-rise data is shown in figure 12. Good agreement was obtained at the lower heating rates. The material property values used in the calculations can be used to make preliminary-design thickness calculations for aerospace applications, provided the flight environment is similar to the lower-heating-rate test conditions.

The calculated surface temperatures are shown in figure 13 as functions of cold-wall heating rate. Specimen 1, which receded about 0.43 cm in 12 000 seconds, had a surface temperature of about 750 K. The higher emittance of the coated specimen gave a surface temperature about 50 K lower. The coated specimen (specimen 8) had about the same surface temperature at a heating rate of 20.4 kW/m^2 as the uncoated specimen (specimen 1) had at a heating rate of 16 kW/m^2. Because specimen 8 was much less degraded than specimen 1 (fig. 8), the coating must not only have increased the surface emittance but also provided resistance to both oxidation and mechanical erosion.

CONCLUDING REMARKS

A low-density (64 kg/m^3), polyimide foam material was tested in an arc tunnel to determine its potential for heat-shield application on aerospace vehicles. An uncoated specimen receded 0.43 cm when tested at a calculated surface temperature of about 750 K for 12 000 seconds. One specimen was coated with a black refractory paint, which raised the surface emittance and toughened the surface. This specimen showed negligible surface recession after being tested at calculated surface temperatures of about 700 K and 750 K for 12 000 and 6000 seconds, respectively.

The test results were compared to values obtained with a computer program. An analytical thermal conductivity for the material was obtained which agreed fairly well with the measured conductivity and provided good agreement between measured and calculated back-surface temperature-rise data. The material properties used in the calculations can be used to make preliminary-design thickness calculations for aerospace applications.

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November 4, 1977

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TABLE I.- TEST ENVIRONMENTS

Test conditions	1	2	3	4	5
Cold-wall heating rate (294 K), kW/m^2	16.0	20.4	25.2	30.8	38.5
Total enthalpy, MJ/kg	1.93	2.25	2.61	3.02	3.55
Local pressure at surface, atm[a]	0.0067	0.0075	0.0080	0.0085	0.0090
Dynamic pressure, atm[a] . . .	0.02	0.02	0.02	0.02	0.02

[a] 1 atm = 101 kPa.

TABLE II.- TEST MATRIX

Specimen	Surface	Test stream	Test condition	No. of cycles	Thickness and mass measured			
					Pretest	After 1 cycle	After 5 cycles	After 10 cycles
1	Uncoated	Air	1	10	X	X	X	X
2	↓	↓	2	5	X	X	X	
3			3	5	X	X	X	
4			4	1	X	X		
5			5	1	X	X		
6			5	1	X	X		
7	↓	Nitrogen	2	5	X	X	X	
8	Coated	Air	1	10	X	X	X	X
8	Coated	Air	2	5	X	X	X	

TABLE III.- TEST RESULTS

Specimen	Test condition	Total recession, percent			Mass loss, percent			Average back-surface temperature rise, K		
		Cycle			Cycle			Cycle		
		First	Fifth	Tenth	First	Fifth	Tenth	First	Fifth	Tenth
1	1	3.2	9	16.5	5.6	14.5	18.7	41	42	44
2	2	4.1	13.8		6.8	17.2		48	53	
3	3	10	32		12	33		69	76	
4	4	18			19			89		
5	5	26			29			81		
6	5	28			29			79		
[a]7	2	3	8.5		5.5	11		59	56	
[b]8	1	2	2	2	3	3	3	34	35	35
[b]8	2	2	2		4	5		44	44	

[a]Tested in nitrogen.

[b]Coated specimen.

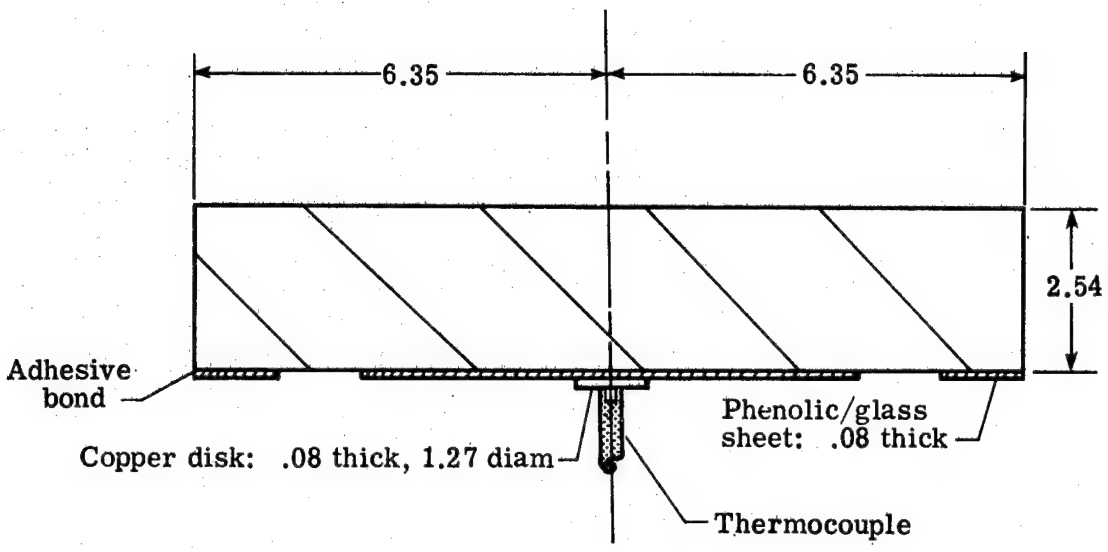

Figure 1.- Section of typical test specimen. Dimensions are in cm.

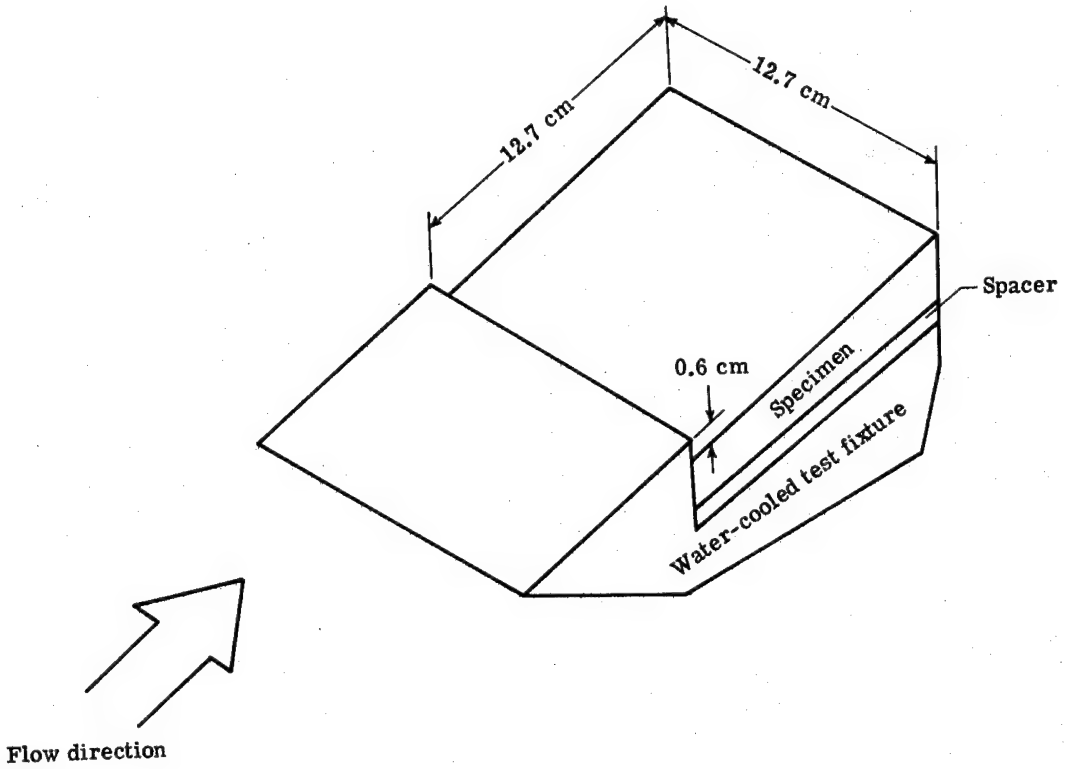

Figure 2.- Arc-tunnel test-specimen configuration.

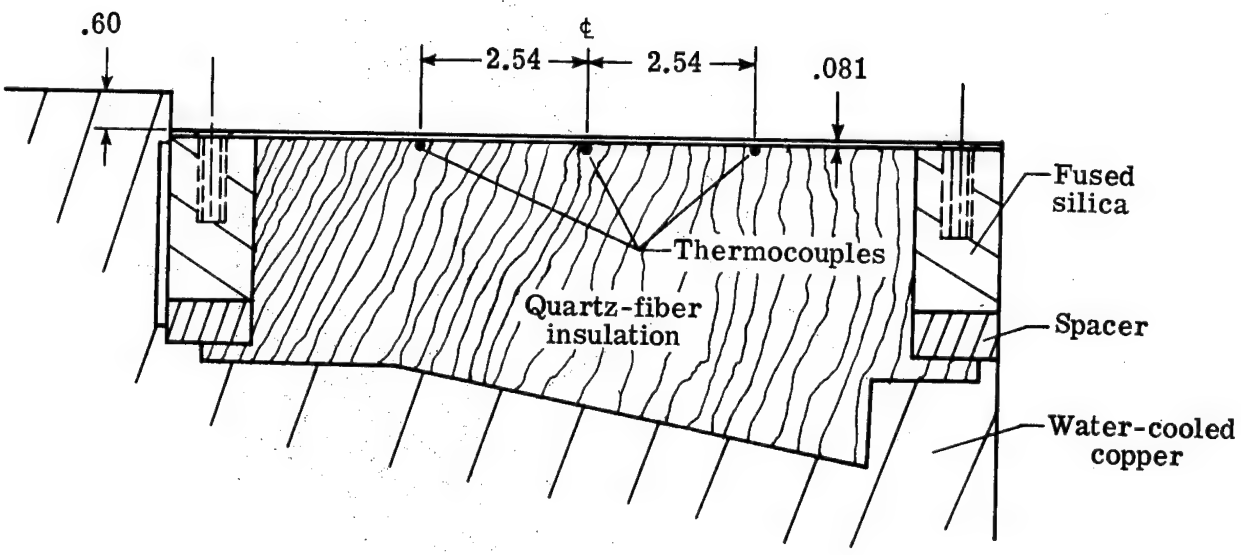

Figure 3.- Arc-tunnel calorimeter configuration. Dimensions are in cm.

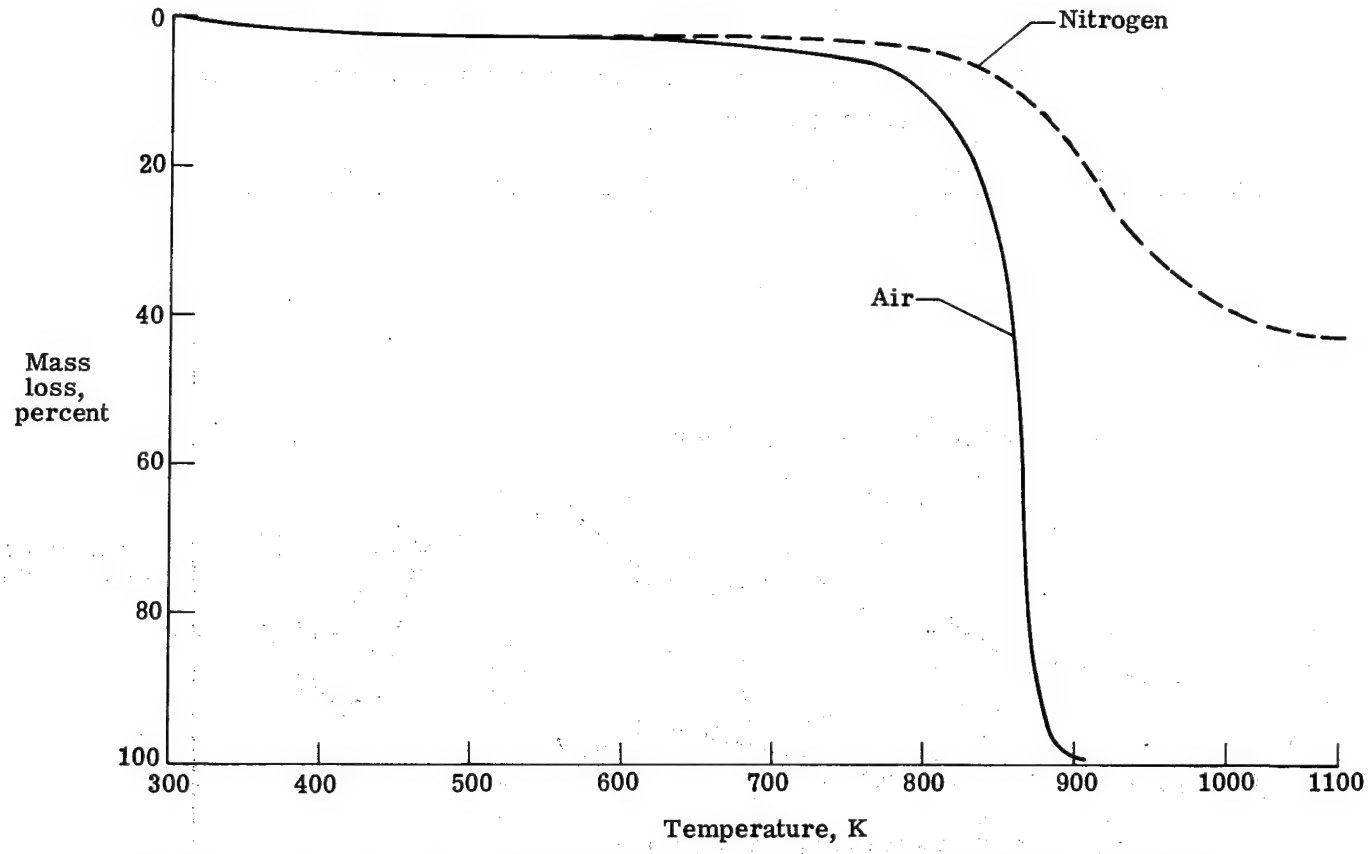

Figure 4.- TGA curve for polyimide foam. Temperature-rise rate = 2.5 K/min.

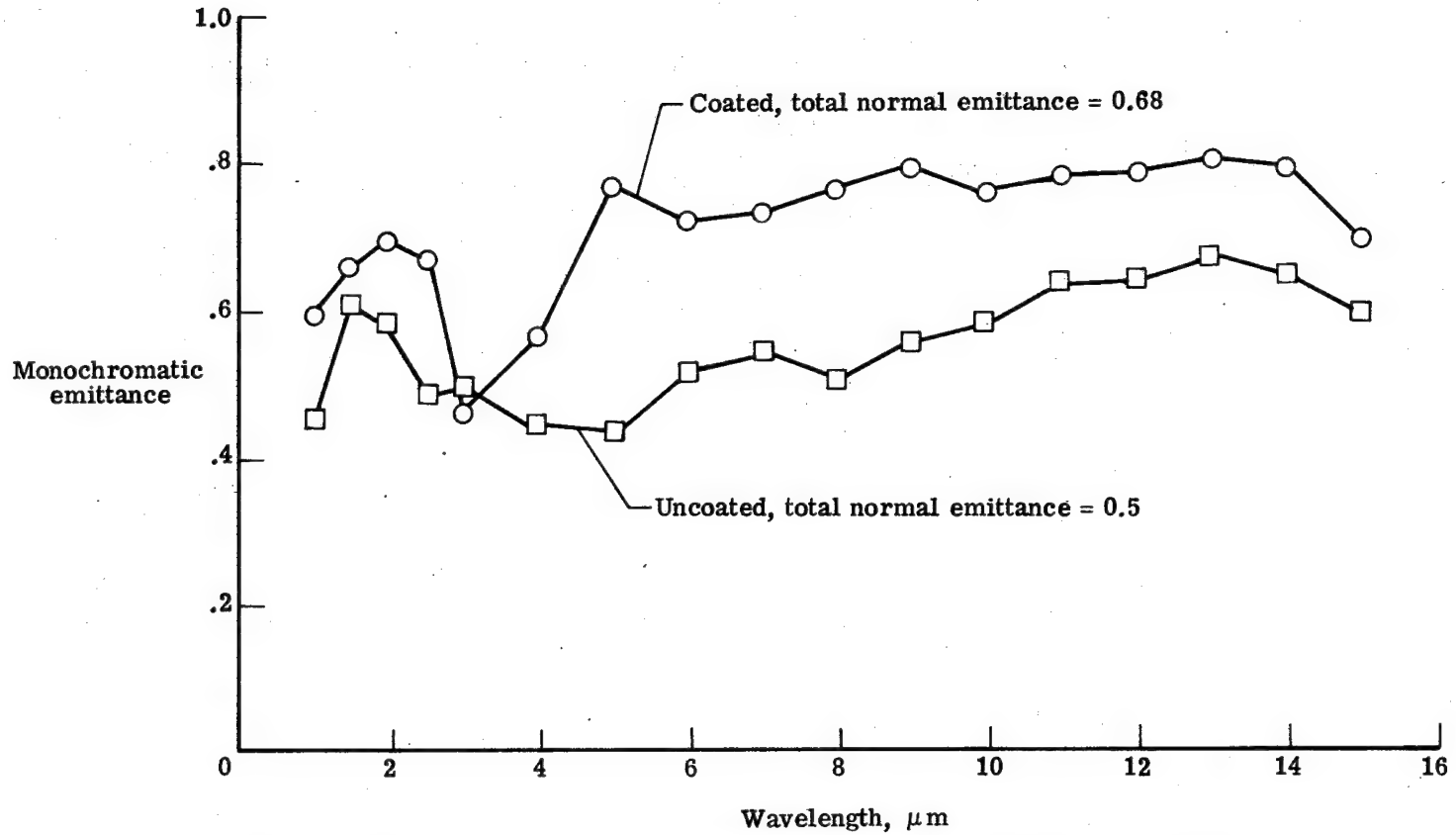

Figure 5.- Emittance spectrum of polyimide foams. Temperature = 700 K.

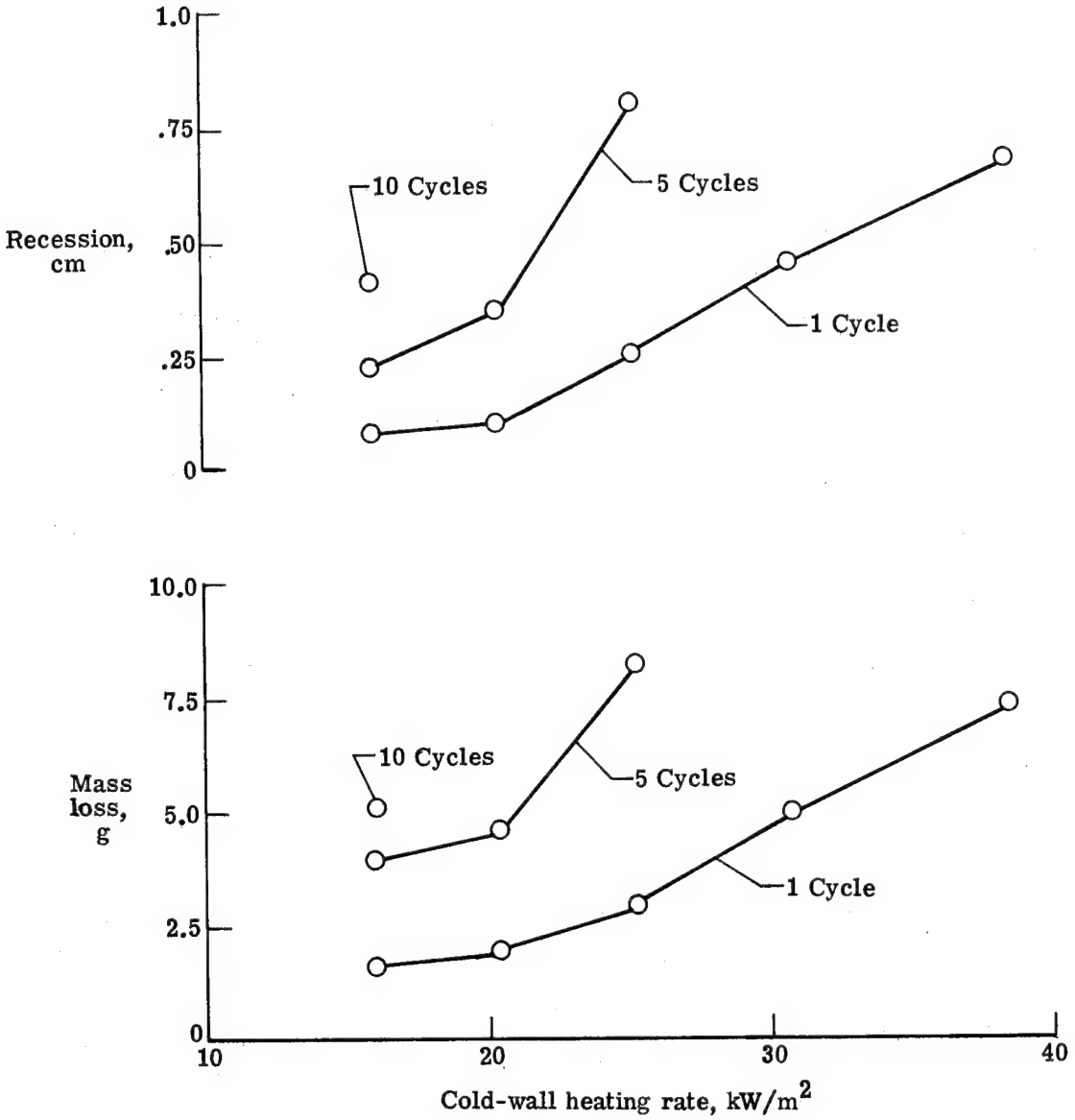

Figure 6.- Recession and mass loss for uncoated specimens tested in air.

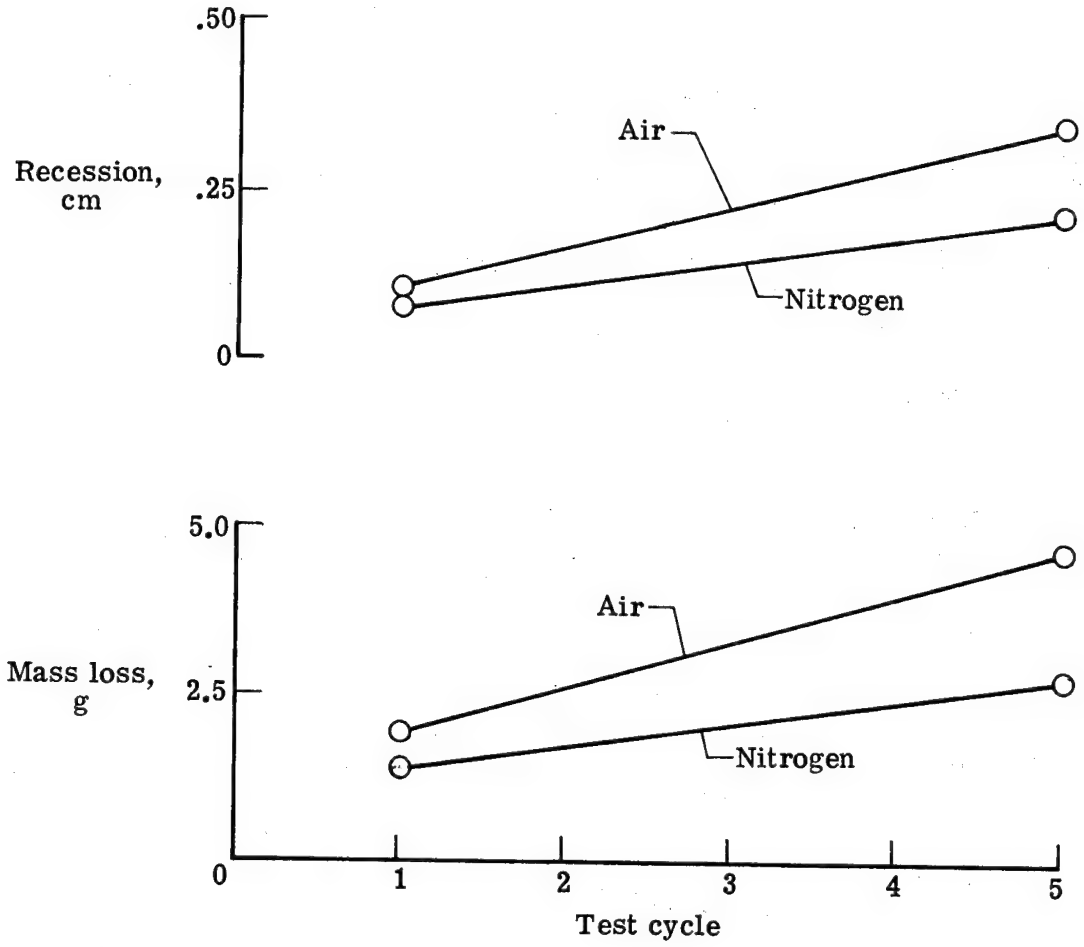

Figure 7.- Effect of oxidation on recession and mass loss. Heating rate = 20.4 kW/m^2.

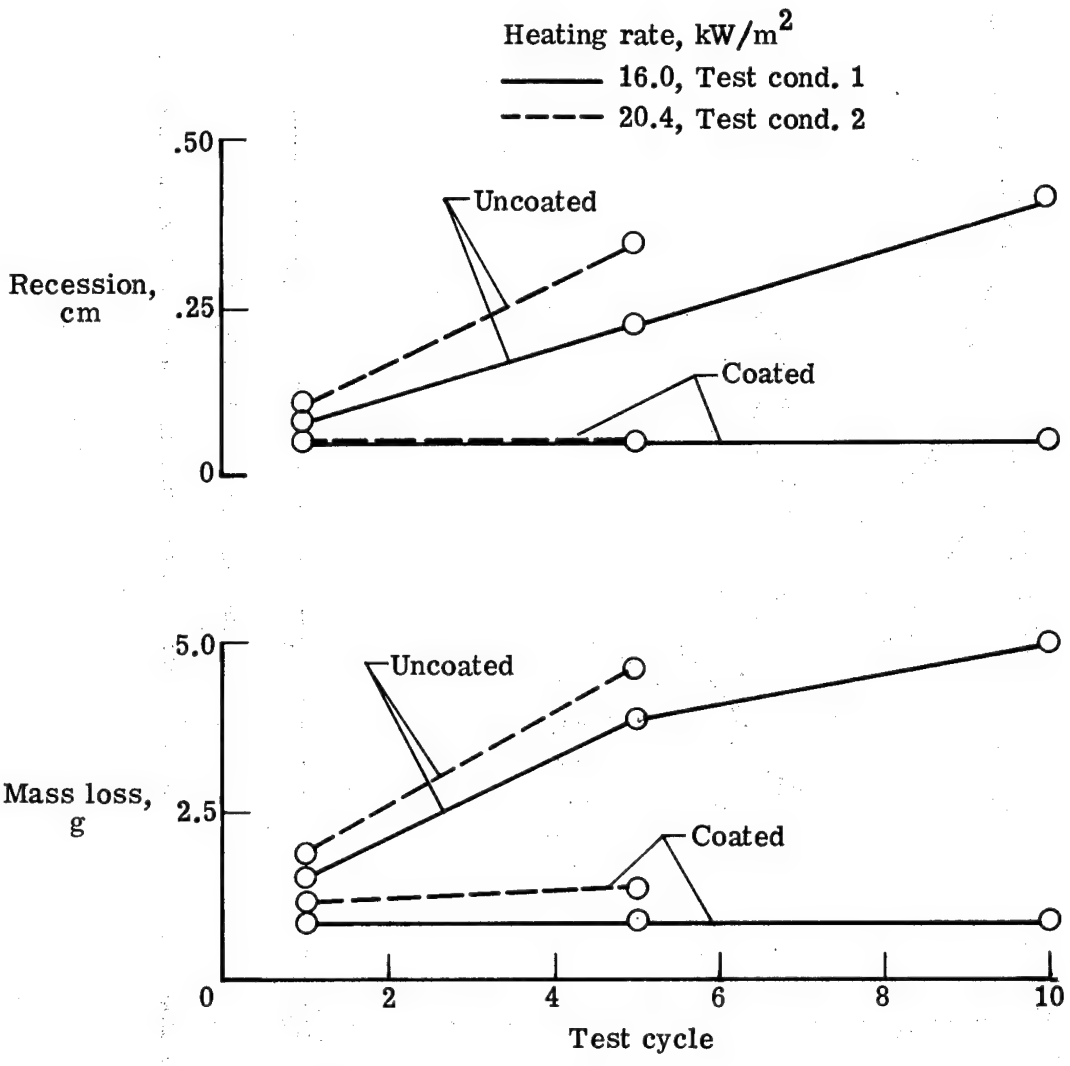

Figure 8.- Effect of coating on recession and mass loss.

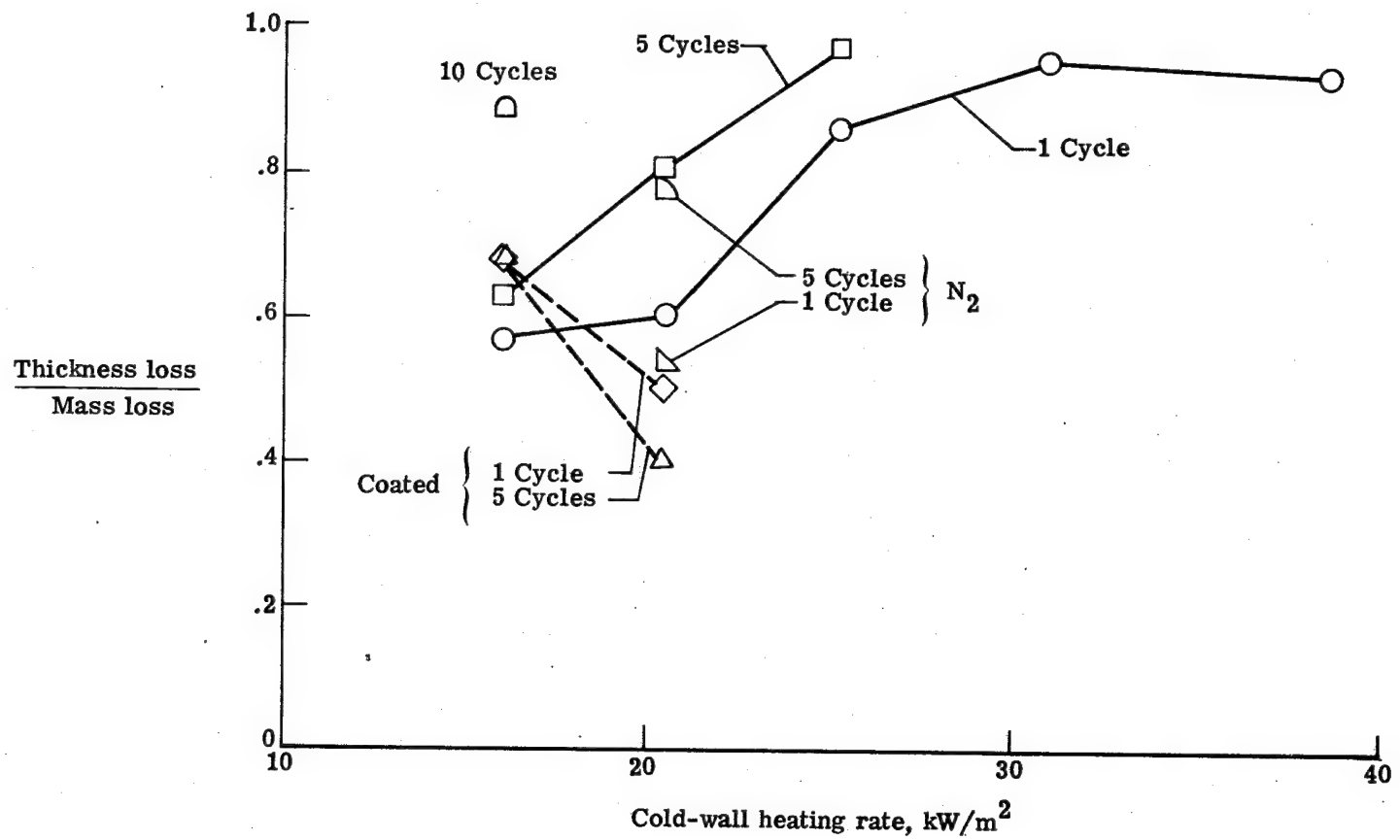

Figure 9.- Change in thickness-loss to mass-loss ratio as function of cold-wall heating rate and number of cycles.

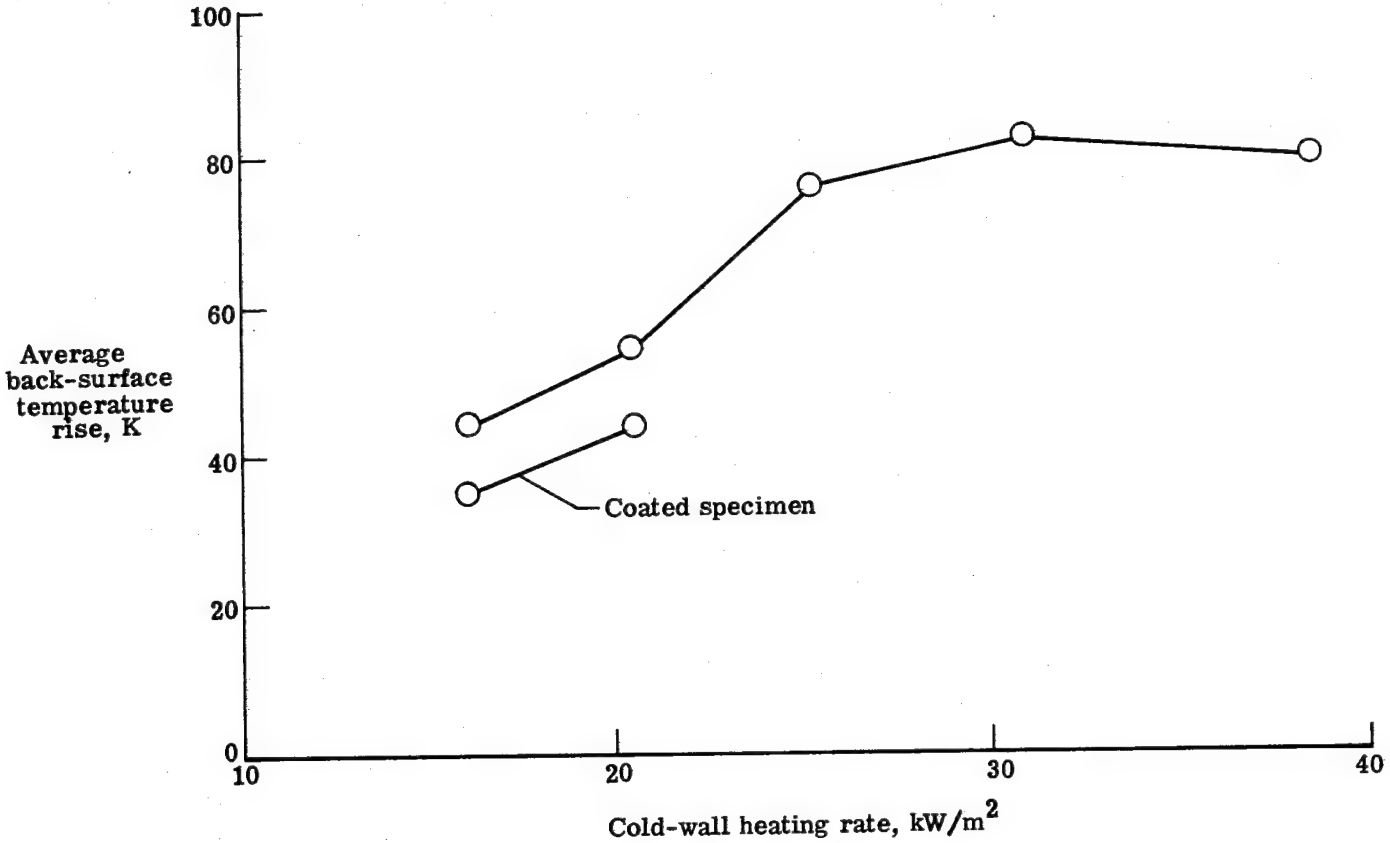

Figure 10.- Average back-surface temperature rise of polyimide-foam specimens as function of cold-wall heating rate.

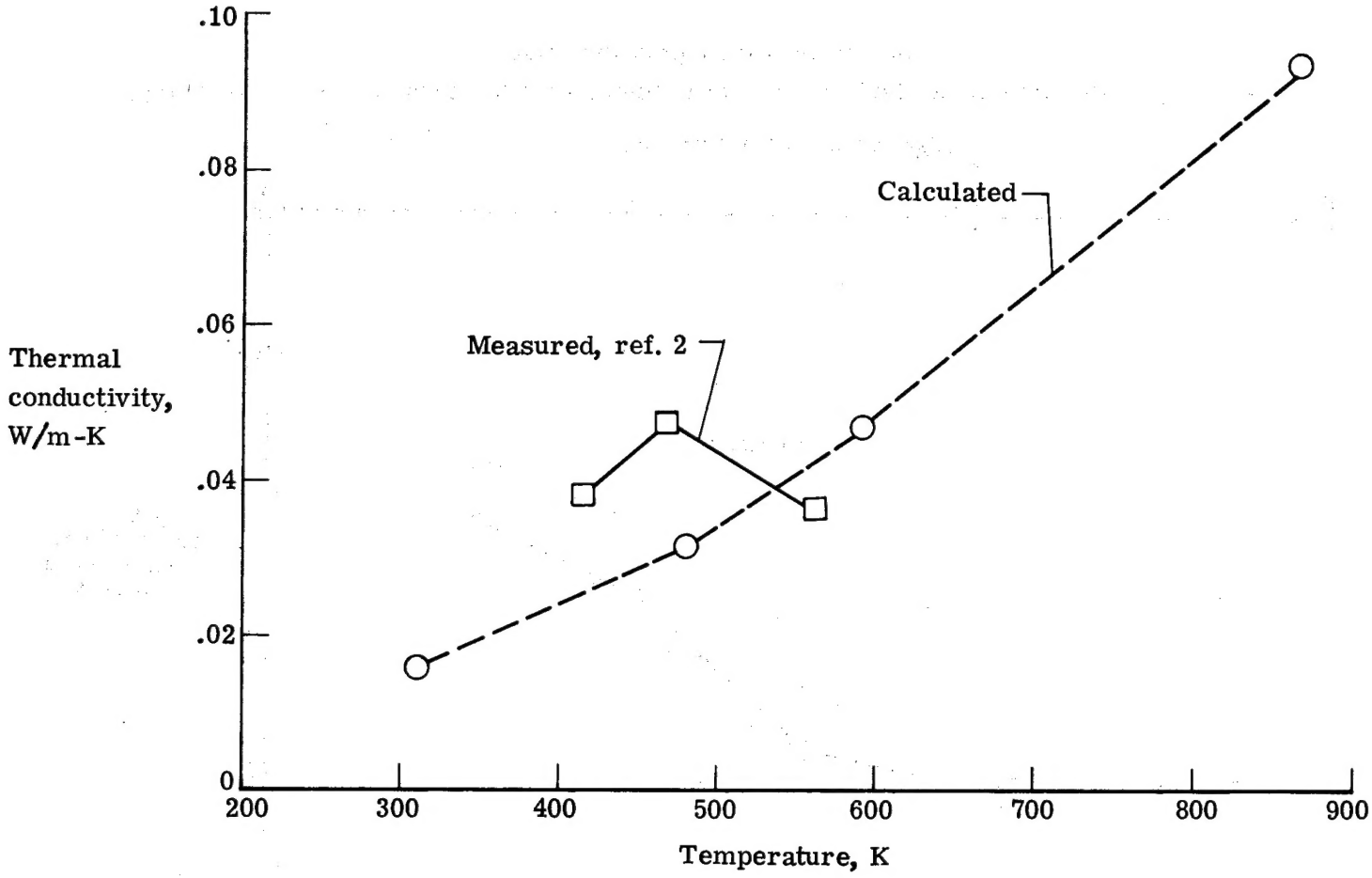

Figure 11.- Comparison of measured and calculated polyimide-foam thermal conductivity.

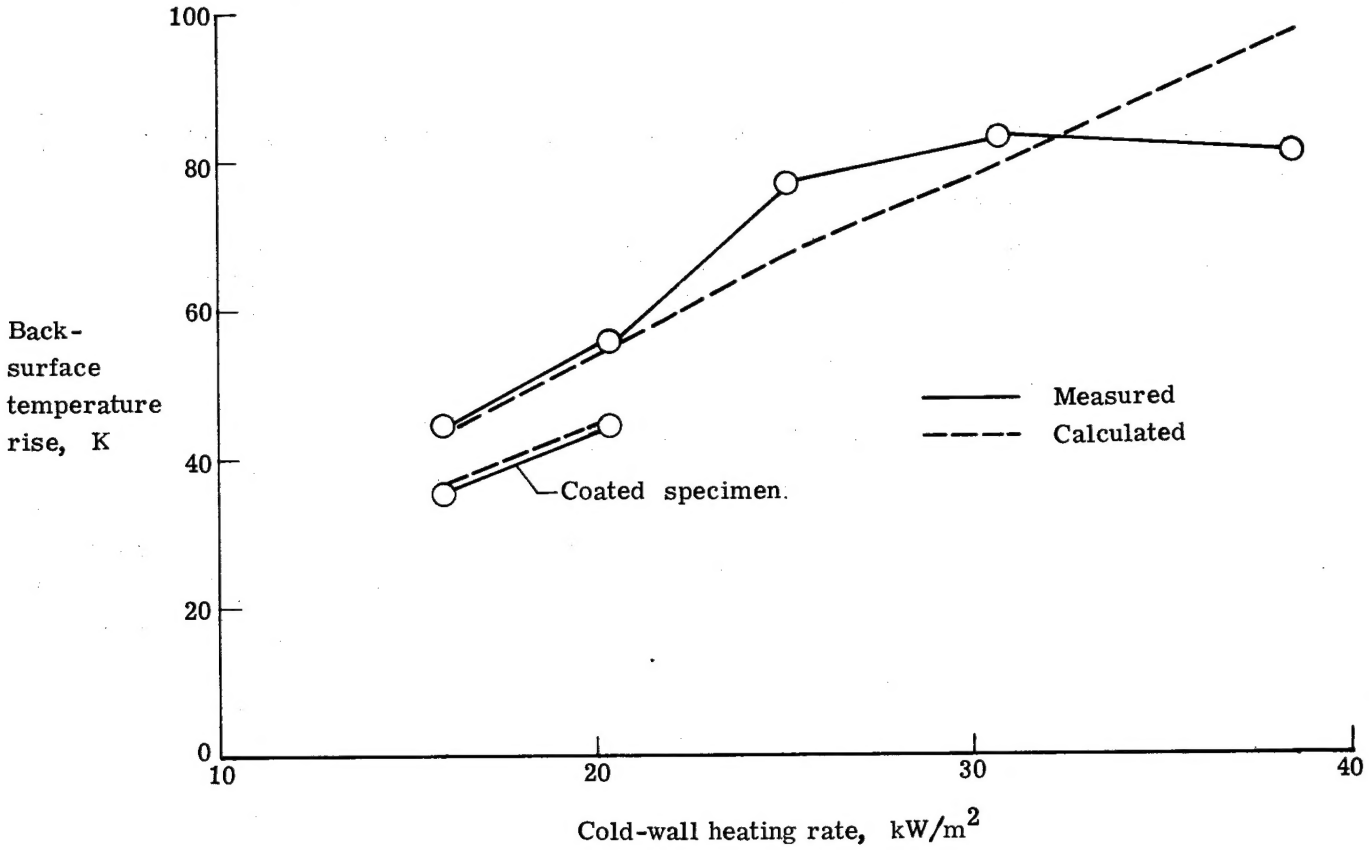

Figure 12.- Comparison of measured and calculated polyimide-foam back-surface temperature rise.

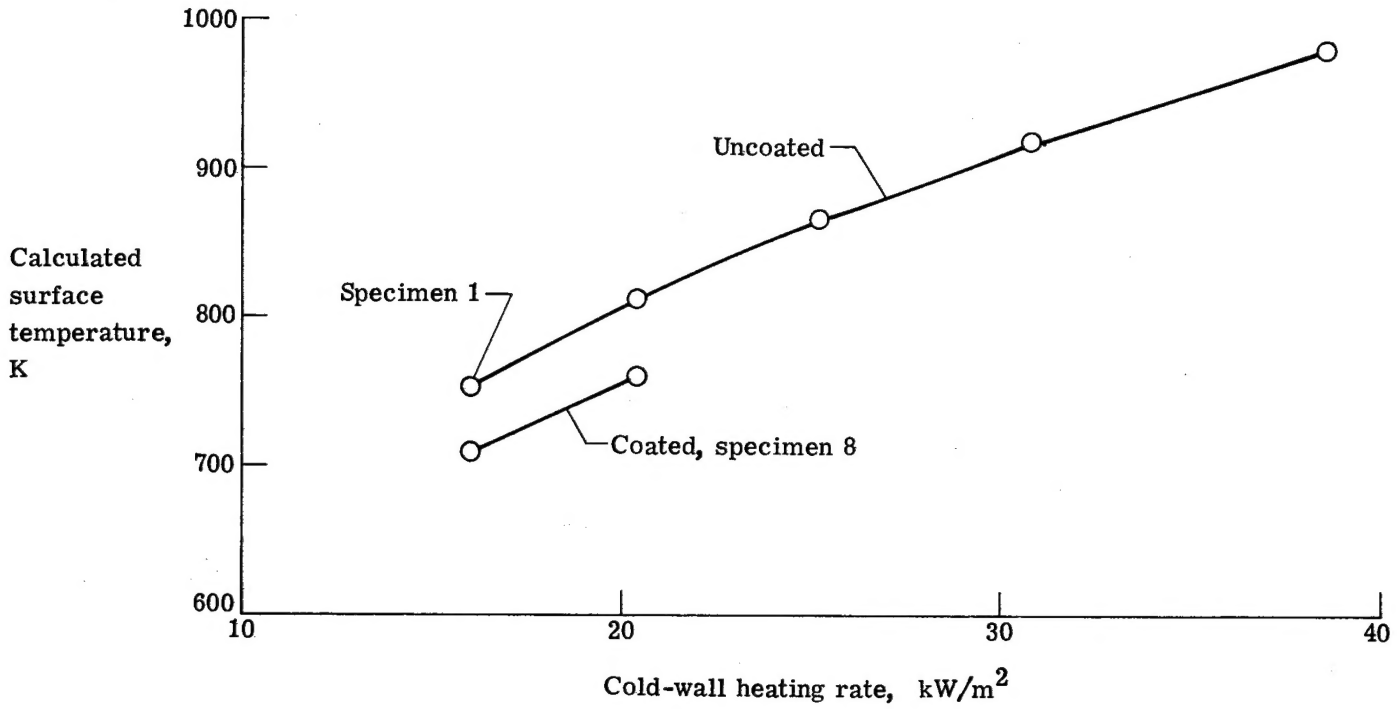

Figure 13.- Calculated surface temperature as function of cold-wall heating rate.

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